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IS 13658 (1993): Polyglycerol Esters of Fatty Acids, Food Grade [FAD 8: Food Additives]

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Indian Standard

POLYGLYCEROL ESTERS OF FATTY ACIDS, FOOD GRADE — SPECIFICATION

UDC 544·099·6 : 547·475·124

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1993 Price Group 3

AMENDMENT NO. 1 FEBRUARY 2006
TO
IS 13658 : 1993 POLYGLYCEROL ESTERS OF FATTY ACIDS, FOOD GRADE — SPECIFICATION

(*Page* 1, *clause* 2.1) — Substitute '1699 : 1995 Methods of sampling and test for food colours (*second revision*)' *for* '1699 : 1974 Methods of sampling and test for food colours (*first revision*)'.

(*Page* 1, *clause* 3.2.3, *line* 5) — Substitute 'isopropanol : water, 90 : 10 (*v/v*)' *for* 'isopropanol : water 90 : 1 (*v/v*)'.

[*Page* 1, *Table* 1, *Sl No.* (vi), *col* 4] — Substitute '15 of IS 1699 : 1995' *for* '10 of IS 1699 : 1974'.

(*Page* 1, *Table* 1) — Delete Sl No. (viii) and renumber the subsequent serial numbers.

[*Page* 1, *Table* 1, *Sl No.* (ix), *col* 4] — Substitute '15 of IS 1699 : 1995 and 18 of IS 6854 : 1973' *for* '11 of IS 1699 : 1974 and 18 of 6854 : 1973'.

(*Page* 2, *clause* 5.1, *line* 5) — Substitute '4 of IS 1699 : 1995' *for* '3 of IS 1699 : 1974'.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

Food Additives Sectional Committee, FAD 8

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

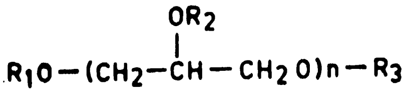
With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties and in some cases to enhance the nutritive values of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have strict quality control of these food additives. A series of standards is, therefore, being prepared by the Bureau to cover purity and identification of these substances. These standards would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity, once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies.

Polyglycerol esters of fatty acids also known as polyglycerol fatty acid ester and glyceran fatty acid esters are the esters of fatty acids of food fats with a mixture of polyglycerols. They may also be prepared by the transesterification of a mixture of polyglycerols with edible fats in which case the product may contain residual mono and diglycerides.

Polyglycerol esters of fatty acids are being permitted for use as release agents in bakery industry and in chocolate manufacture.

Structural Formula

The major components have the following general formula;



$$R_1O-(CH_2-\overset{\displaystyle OR_2}{\overset{|}{C}H}-CH_2O)n-R_3$$

where the average value of *n* is about 3 and R_1, R_2 and R_3 each may be a fatty acid moiety or hydrogen.

In the preparation of this standard due consideration has been given to the provisions of the *Prevention of Food Adulteration Act, 1954* and the Rules framed thereunder and the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977*. However, this standard is subject to the restrictions imposed under these, wherever applicable.

This standard is based on the EEC Directive No. 78/663/EEC laying down specific criteria of purity for emulsifiers, stabilizers, thickners and gelling agents for use in foodstuffs. Considerable assistance has also been derived from the following publications:

Food and Nutrition Paper No. 4 'Specification for identity and purity of thickening agents, anticaking agents, antimicrobials, antioxidants, emulsifiers'. Food and Agriculture Organization of the United Nations, Rome 1978.

Food Chemical Codex, 1981 Pub. National Academy of Sciences and National Research Council, Washington DC, USA.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POLYGLYCEROL ESTERS OF FATTY ACIDS, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for polyglycerol esters of fatty acids.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
548 (Part 1) : 1964	Method of sampling and test for oils and fats: Part 1 Methods of sampling, physical and chemical tests
1070 :1992	Reagent grade water (*third revision*)
1699 :1974	Methods for sampling and tests for food colours (*first revision*)
6854 :1973	Method of sampling and test for ingredients used in media for microbiological work
11912 :1986	Lime oil, distilled, food grade

3 REQUIREMENTS

3.1 Description

Polyglycerol esters of fatty acids are yellowish to amber unctuous liquids, semi-solids or waxy solids.

3.2 Identification Tests

3.2.1 *Solubility*

The esters range from very hydrophilic to very lipophilic but as a class tend to be dispersible in water and soluble in organic solvents and oils.

3.2.2 The product shall give a positive test for fatty acids when determined by the method of test given in Annex A.

3.2.3 Spot 5 to 20 µl of the aqueous layer obtained in A-1.1.1 alongside control spots of glycerol on paper such as Whatman No. 3 and develop using descending chromatography for 36 hours with isopropanol : water 90 : 1 (*v*/*v*). The glycerol spot moves 40 cm and the polyglycerols are revealed in succession below that for glycerol when the paper is sprayed with either permanganate in acetone or ammoniacal silver nitrate.

3.3 Purity Tests

3.3.1 *Acids*

Acids other than fatty acids shall not be detectable when tested by the method given in Annex B.

3.3.2 *Polyglycerols*

The polyglycerol moiety shall be composed of not less than 75 percent of di, tri and tetraglycerols and shall contain not more than 10 percent of polyglycerols equal to or higher than heptaglycerol when tested by the method given in Annex C.

3.3.3 The materials shall also conform to the requirements given in Table 1.

Table 1 Requirements for Polyglycerol Esters of Fatty Acids, Food Grade

Sl No.	Characteristics	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Total fatty acid ester content, percent by mass, *Min*	90	Annex D
ii)	Free fatty acids (estimated as oleic acid), percent by mass, *Max*	6	7 of IS 548 (Part 1) : 1964
iii)	Total glycerol and polyglycerol, percent by mass	18-60	Annex C and E
iv)	Free glycerol and polyglycerol, percent by mass, *Max*	7	Annex C and F
v)	Sulphated ash, percent by mass, *Max*	0.5	Annex G
vi)	Arsenic (as As), mg/kg, *Max*	3	10 of IS 1699 : 1974
vii)	Heavy metals (as Pb), mg/kg, *Max*	10	Annex B of IS 11912 : 1986
viii)	Lead (as Pb), mg/kg, *Max*	10	9 of IS 1699 : 1974
ix)	Copper and Zinc, mg/kg, *Max*	50	11 of IS 1699 : 1974 and 18 of 6854:1973
x)	Zinc, mg/kg, *Max*	25	18 of IS 6854 : 1973

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be securely packed in well-filled containers with minimum access to light and moisture. The containers shall be such as to preclude contamination of the contents with metal or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to aviod excessive exposure to heat.

4.3 Marking

Each container shall be legibly marked to give the following information:

a) Name of the material including the words 'food grade';
b) Source of manufacture;
c) Minimum net mass of the contents;
d) Batch or code number;
e) Date of manufacture; and
f) Any other details required as per the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977/PFA Rules.

4.3.1 The container may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined according to the procedure prescribed in 3 of IS 1699 : 1974.

6 TESTS

6.1 Tests shall be carried out by the methods as specified in 3.2.2, 3.2.3, 3.3 and col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

ANNEX A

(*Clause* 3.2.2)

TEST FOR FATTY ACIDS

A-1 PROCEDURE

A-1.1 Reflux 1 g of sample with 15 ml of 0.5 N ethanolic potassium hydroxide for 1 hour. Add 15 ml of water, acidify with about 6 ml of dilute hydrochloric acid. Oily drops or a white to yellowish white solid is produced which is soluble in 5 ml of hexane.

A-1.1.1 Remove the hexane layer. Extract again with 5 ml of hexane and again remove the hexane layer. Use the aqueous layer for detection of acids other than fatty acids.

ANNEX B

(*Clause* 3.3.1)

DETECTION OF ACIDS OTHER THAN FATTY ACIDS

B-1 PROCEDURE

B-1.1 Acetic Acid

B-1.1.1 Transfer about 5 ml of the aqueous layer (*see* A-1.1.1) into a dish. Add excess calcium carbonate and evaporate until dry. Transfer the major part of the residue into a glass tube. Place a filter paper, moistened with reagent for acetone (a saturated solution of o-nitrobenzaldehyde in sodium hydroxide, freshly prepared) on the top of the tube. Heat as indicated in Fig. 1.

The yellow colour of the paper changes into greenish blue by reaction of the reagent for acetone, with the calcium acetate formed.

B-1.2 Succinic Acid

B-1.2.1 Transfer one drop of the aqueous layer (*see* A-1.1.1) and a drop of a 0.5% solution of ammonium chloride and several mg of zinc powder into a micro test tube.

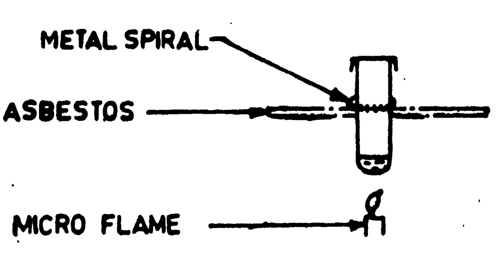


FIG. 1 HEATING ARRANGEMENT

B-1.2.2 The mouth of the tube is covered with a disk of filter paper moistened with a solution, in benzene, of 5% p-dimethylaminobenzaldehyde and 20% trichloroacetic acid. The bottom of the test tube is heated vigorously with a micro flame (Fig.1) for about 1 minute. Depending on the amount of succinic acid or succinimide, a red-violet or pink stain appears on the paper.

B-1.3 Fumaric Acid

Transfer 1 ml of the aqueous layer (*see* A-1.1.1) with 1 ml of 2 *N* sodium carbonate into a test tube. Add 2 or 3 drops of 0.1 *N* potassium permanganate. The solution is promptly discoloured.

B-1.4 Tartaric Acid

Evaporate about 5 ml of the aqueous layer (*see* A-1.1.1) in a porcelain dish until dry. Add 2 ml of concentrated sulphuric acid containing 0.5% of pyrogallol and heat on a steam bath. An intense violet colour is produced.

B-1.5 Citric Acid

B-1.5.1 To 3 ml of the aqueous layer (*see* A-1.1.1) add a few drops of 1% potassium permanganate and warm until the colour has disappeared. Then add an excess of bromine solution. A white precipitate (pentabromoacetone) is formed immediately on cooling.

B-1.5.2 Evaporate 1 ml of the aqueous layer resulting from test in a porcelain dish, add 1 ml of a mixture of 1 volume acetic anhydride and 5 volume of pyridine into the warm dish. A violet colour is produced. (Tartaric acid produces a green colour.)

B-1.6 Lactic Acid

B-1.6.1 Transfer 0.2 ml of the aqueous layer (*see* A-1.1.1) add 2 ml of concentrated sulfuric acid into a test tube and place for 2 minutes in boiling water. Cool and add 1 or 2 drops of a 5% guaiacol solution in ethanol. A red colour is immediately produced.

B-1.6.2 If tartaric acid is present according to test 5, it must be removed as follows:

> Transfer 3 ml of the aqueous layer (*see* A-1.1.1) and an excess of calcium hydroxide as a powder into a test tube, place in boiling water for 5 minutes, shaking several times, cool and filter.

ANNEX C

[*Clause* 3.3.2, *Table* 1, *Sl No.* (iii) *and* (iv)]

ANALYSIS OF POLYOLS IN POLYGLYCEROL ESTERS OF FATTY ACIDS

C-1 PRINCIPLE

Polyglycerol esters are saponified with alcoholic potassium hydroxide solution and the fatty acids removed by extraction. The polyols are converted to trimethylsilyl (TMS) derivatives and analysed by gas liquid chromatography.

C-2 PROCEDURE

C-2.1 Preparation of the Polyol Sample

Weigh about 0.5 g of sample and reflux with 20 ml of ethanolic potassium hydroxide solution (1*N*) for 2 hours. Reduce the volume of ethanol by evaporation at 45-50° in a stream of nitrogen. Add 10 ml of water and convert the soaps to free fatty acids by acidifying with concentrated hydrochloric acid. Extract the fatty acids from the aqueous phase with successive 20 ml portions of light petroleum extracts with water (20 ml) and combine the wash with the aqueous phase.

Adjust the aqueous polyol solution to *p*H 7.0 with aqueous potassium hydroxide solution with the aid of a *p*H-meter. Evaporate to a small volume (2-3 ml) under reduced pressure and extract three times with 30 ml of boiling ethanol. Filter off any residue and evaporate the ethanol under reduced pressure to yield a viscous liquid sample of polyols.

Dissolve a 0.1 g sample of polyol in 0.5 ml of warm pyridine (previously dried over potassium hydroxide) in a 10 ml capped phial. Add 0.2 ml hexamethyl disilazane, shake, add 0.2 ml trimethylchlorosilane and shake again. Stand on a warm plate (about 80°) for 3-5 minutes. Check that white fumes are present indicating an excess of reagent.

C-2.3 Gas-Liquid Chromatography

C-2.3.1 Any suitable gas chromatograph may be used equivalent to a Pye Model 104 fitted with a flame ionization detector and a column (1.5 m x 4 mm I.D.) packed with 3% OVI on Diatomite CO (100-120 mesh) or on Gas Chrom 0 (100-120 mesh). Recommended conditions are : *oven temperature*, programmed from 90° to 330° at 4-6°/min; *nitrogen carrier gas flow rate*, 86 ml/min; *injection block temperature*, 275°; *detector block temperature*, 350°.

Inject a 2.0 µl sample of TMS derivatives of polyols. The resultant chromatograph displays the following sequence of peaks:

Elution Sequence of Peaks	*Identity*	*Description (and Typical Attenuation Settings)*
1.	Solvent	Overloaded
2.	Glycerol	Single peak (2 x 10^3)
3.	Cyclic diglycerols	Single peak (2 x 10^3)
4.	Diglycerols	Single peak (32 x 10^3)

Elution Sequence of Peaks	*Identity*	*Description (and Typical Attenuation Settings)*
5.	Cyclic triglycerols	Single peak (2×10^3)
6.	Triglycerols	Single peak (16×10^3)
7.	Cyclic tetraglycerols	Single peak (2×10^3)
8.	Tetraglycerols	Multiple peak (8×10^3)
9.	Pentaglycerols	Single peak (4×10^3)
10.	Hexaglycerols	Single peak (2×10^3)
11.	Heptaglycerols	Single peak (2×10^3)
12.	Octaglycerols	Single peak (1×10^3)
13.	Nonaglycerols	Barely discernible in the tail of peak 12.

C-2.3.3 *Calculation*

Measure each peak area by a suitable method and correct for attenuation changes.

% di-, tri- and tetraglycerols

$$= \frac{\text{Sum of corrected areas of peaks 3 to 8} \times 100}{\text{Sum of corrected areas of peaks 3 to 13}}$$

% polyglycerols equal to or greater than heptaglycerol

$$= \frac{\text{Sum of corrected areas of peaks 11 to 13} \times 100}{\text{Sum of corrected areas of peaks 3 to 13}}$$

ANNEX D

[*Table 1, Sl No.* (i)]

DETERMINATION OF TOTAL FATTY ACID ESTERS CONTENT

D-0 PRINCIPLE

A distribution of the starting material between a chloroform and a water phase is established, and the fatty acids are extracted by chloroform, and estimated after distilling off the chloroform.

D-1 REAGENTS

D-1.1 Chloroform

D-1.2 Methanol

D-1.3 Magnesium Chloride Solution in Water —1 Molar.

D-2 APPARATUS

D-2.1 Erlenmeyer Flask — 1 litre

D-2.2 Separating Funnel — 1 litre

D-2.3 Separating Funnel — 500 ml

D-2.4 Round-Bottomed Flask — 500 ml

D-2.5 Magnetic Stirrer

D-2.6 Rotation Evaporator

D-2.7 Water Jet Pump

D-2.8 Heating Cupboard

D-2.9 Desiccator

D-3 PROCEDURE

D-3.1 Take 20 g of a homogenous sample of emulsifier in a 1-litre Erlenmeyer flask. Dissolve in 100 ml chloroform. Add to it 200 ml methanol and mix the blend with a magnetic stirrer. After mixing, add another 100 ml chloroform and continue the mixing. Transfer the solution into a 1-litre separating funnel. Wash the Erlenmeyer flask with 30 ml chloroform and transfer to the separating funnel. Then add 180 ml water and mix the contents of the separating funnel. Let the two phases separate from each other. Extract chloroform phase two times with a 10-ml mixture of 9 : 10 — water : methanol. After separating, the water : methanol mix is combined with the original water phase. Wash the combined water phase, once, in a 500-ml separating funnel with 50 ml chloroform.

D-3.2 Transfer the chloroform phase which contains the fatty acid ester to a 500-ml round bottomed flask and distil off the solvent in a rotational distilling apparatus under vacuum from a water jet pump. After the chloroform has been removed, treat the flask further for 20 minutes at the distilling rotator. Dry the flask for 20 minutes in a heating cupboard at 105°C and after cooling in a desiccator weigh.

NOTE — If an emulsion is formed in the solvent, add 1 ml of a 1 molar magnesium chloride solution.

D-4 CALCULATION

$$\text{Total fatty acid ester content, percent by mass} = \frac{X}{M} \times 100$$

where

M = mass, in g, of material taken for the test; and

X = mass, in g, of the material remaining in the 500-ml round bottomed flask after cooling in the desiccator.

ANNEX E

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF TOTAL GLYCEROL

E-0 PRINCIPLE

The material is saponified with alcoholic potassium hydroxide solution and the water soluble components of the neutralized solutions are purified and determined gravimetrically.

E-1 REAGENTS

E-1.1 Alcoholic Potassium Hydroxide Solution—0.5 N.

E-1.2 Hydrochloric Acid — 0.5 N.

E-1.3 Hexane (for analysis)

E-1.4 2-Propanol (for analysis)

E-2 APPARATUS

E-2.1 Erlenmeyer Flask — 300-ml.

E-2.2 Separating Funnel — 500-ml.

E-2.3 Round-Bottomed Flask — 200-ml, with NS 29.

E-2.4 Round-Bottomed Flask — 100-ml, with NS 29.

E-2.5 Water Bath

E-2.6 Rotation Evaporator

E-2.7 Water Jet Pump

E-2.8 Heating Cupboard

E-2.9 Desiccator

E-2.10 Condenser

E-3 PROCEDURE

The analysis are carried out in duplicate. Weigh 1 g of the polyglycerol ester in a 300-ml Erlenmeyer flask. Add 25 ml of 0.5 N alcoholic potassium hydroxide solution and saponify the sample for 30 minutes by boiling under reflux.

After saponification neutralize, the sample with an equivalent amount of 0.5 N hydrochloric acid. Further add 0.5 ml hydrochloric acid. Distil off the alcohol in the rotating evaporator. Transfer the contents of the flask to a 500-ml separating funnel with 100 ml hexane and 25 ml distilled water. Shake. After the separation, transfer the water phase to a 200-ml flask fitted with NS 29. Wash the hexane phase two times with 25 ml distilled water each time. Both the water phases are united with the first phase in the flask. Distil the water in the rotating evaporator. Dissolve the remaining glycerol in 2- propanol and filter through a Whatman filter paper No. 41 of 9 cm diameter into a 100-ml flask fitted with NS 29. Wash the flask and filters with 2-propanol in order to transfer the sample quantitatively. After distilling off the 2-propanol in the rotating evaporator on a water bath, apply full vacuum from the water jet pump for 20 minutes followed by drying for 20 minutes in the heating cupboard and finally cool in the desiccator. Weigh.

NOTE — The 100-ml round-bottomed flask with NS 29 is first dried at 105°C and after cooling in a desiccator weighed with an accuracy of 0.000 5 g.

E-4 CALCULATION

$$\text{Total glycerol, percent by mass} = \frac{b \times 100}{a}$$

where

b = mass, in g, of the dried polyalcohol phase; and

a = mass, in g, of the ester.

ANNEX F

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF FREE GLYCEROL

F-0 A distrioution of the starting material betwen a chloroform phase and a water phase is established, the free glycerol collected from the water phase and estimated after drying the water phase.

F-1 REAGENTS

F-1.1 Chloroform

F-1.2 Methanol

F-1.3 Magnesium Chloride Solution in Water—1 -Molar

F-2 APPARATUS

F-2.1 Erlenmeyer Flask — 1-litre.

F-2.2 Separating Funnel — 1-litre.

F-2.3 Separating Funnel — 500-ml.

F-2.4 Round-Bottomed Flask — 500-ml.

F-2.5 Magnetic Stirrer

F-2.6 Rotation Evaporator

F-2.7 Water Jet Pump

F-2.8 Heating Cupboard

F-2.9 Desiccator

F-3 PROCEDURE

Proceed as given under D-3.1. Transfer the water phase into a weighed 500-ml round-bottomed flask and distil off the solvent in a rotating evaporator under vacuum from a water jet pump. As soon as the chloroform and the methanol has been distilled off, treat the flask further full vacuum in the rotating evaporator. Dry the flask for 20 minutes in a heating cupboard at 105°C and after cooling in a desiccator, weigh.

NOTE — If there is a tendency to emulsion formation, add 1 ml of a 1 molar magnesium chloride solution.

F-4 CALCULATION

$$\text{Free glycerol, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass, in g, of material remaining in the 500 -ml round- bottomed flask; and

M = mass, in g, of the material taken for the test.

ANNEX G

[*Table* 1, *Sl* No. (v)]

DETERMINATION OF SULPHATED ASH

G-1 DETERMINATION OF SULPHATED ASH

G-1.1 Reagents

G-1.1.1 *Dilute Sulphuric Acid* — 10 percent (*m/v*)

G-1.2 Procedure

Transfer about 2 g of the sample, accurately weighed, to a tared 50-ml to 100-ml platinum dish or other suitable container and add sufficient diluted sulphuric acid to moisten the entire samples. Heat gently, until the sample is dry and thoroughly charred, then continue heating until all the sample has been volatilized or nearly all of the carbon has been oxidized. Cool. Moisten the residue with 0.1 ml of sulphuric acid, and heat in the same manner until the remainder of the sample and any excess sulphuric acid have been volatilized. Finally ignite in a muffle furnace at 800± 25°C for 15 minutes. Cool in a desiccator and weigh.

G-1.3 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass, in g, of residue; and

M = mass, in g, of the material taken for the test.

Standard Mark

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Amendments are issued to standards as the need arises on the basis of comments. Standards are any reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'. Comments on this Indian Standard may be sent to BIS giving the following reference:

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